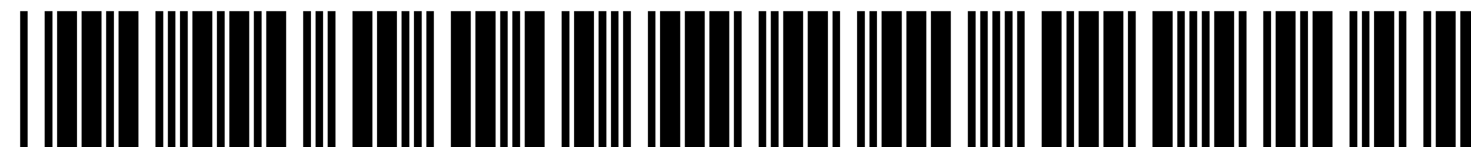
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Shiba

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(54) **METHOD FOR FORMING ALUMINUM NITRIDE-BASED FILM BY PEALD**

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CPC H01L 21/3185; H01L 21/02274; H01L 21/31612; H01L 21/02164; H01L 21/022; H01L 21/0228; H01L 21/76834; H01L 23/53228; H01L 23/53295
See application file for complete search history.

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(57) **ABSTRACT**

A method for forming an aluminum nitride-based film on a substrate by plasma-enhanced atomic layer deposition (PEALD) includes: (a) forming at least one aluminum nitride (AlN) monolayer and (b) forming at least one aluminum oxide (AlO) monolayer, wherein steps (a) and (b) are alternately conducted continuously to form a laminate. Steps (a) and (b) are discontinued before a total thickness of the laminate exceeds 10 nm, preferably 5 nm.

11 Claims, 5 Drawing Sheets

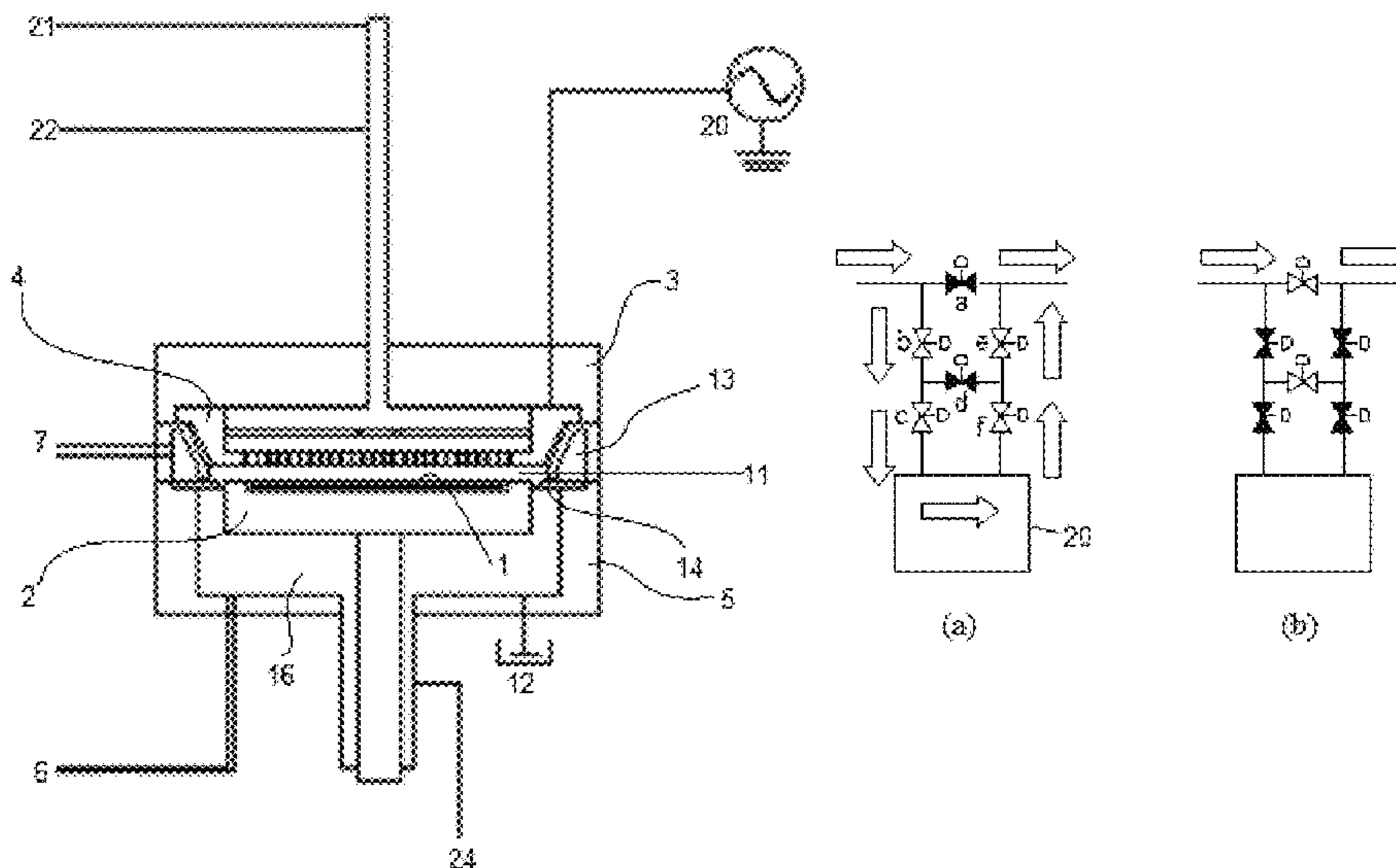

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Fig. 1A

(a)

(b)

Fig. 1B

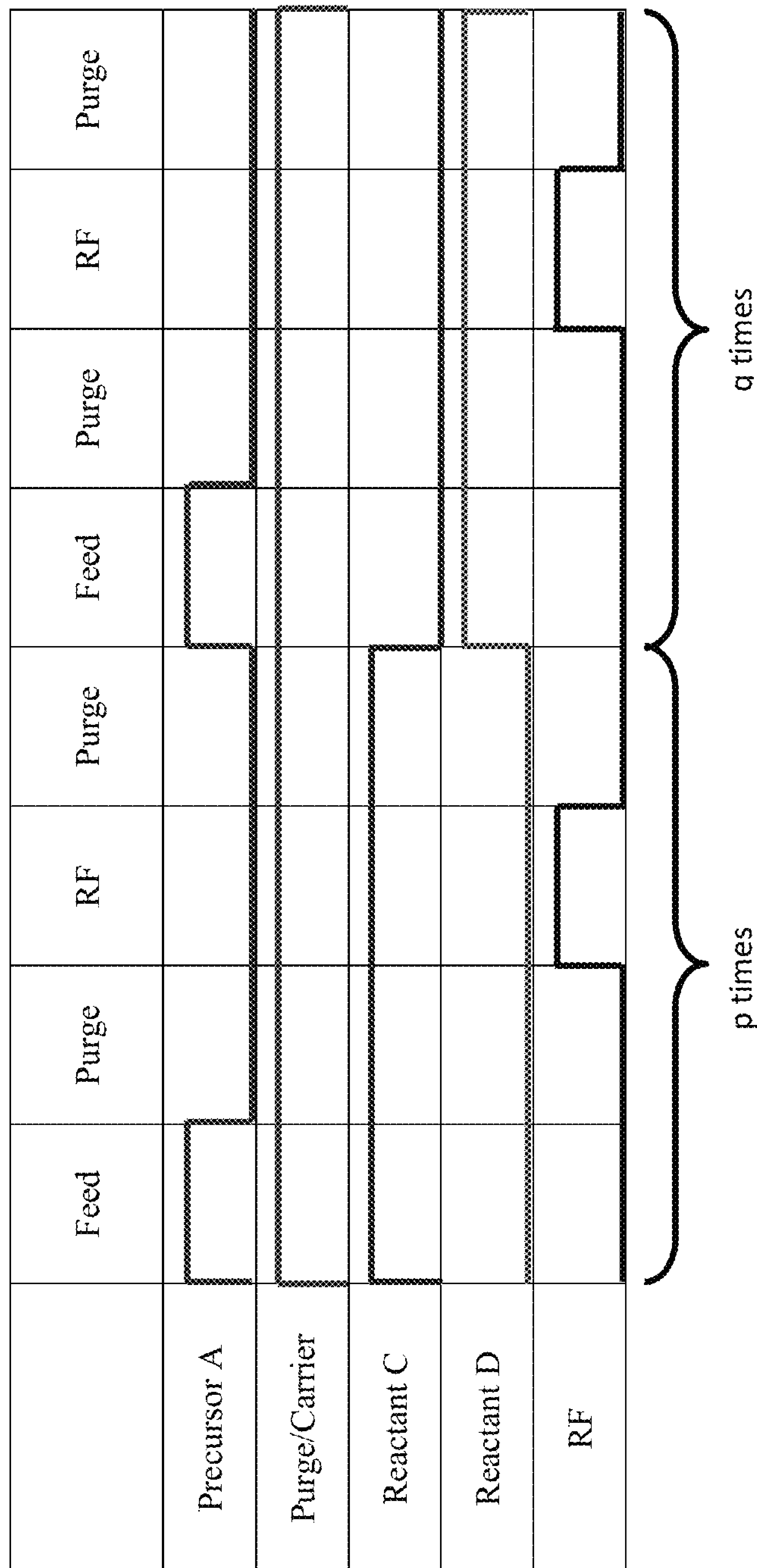


Fig. 2

Fig. 3

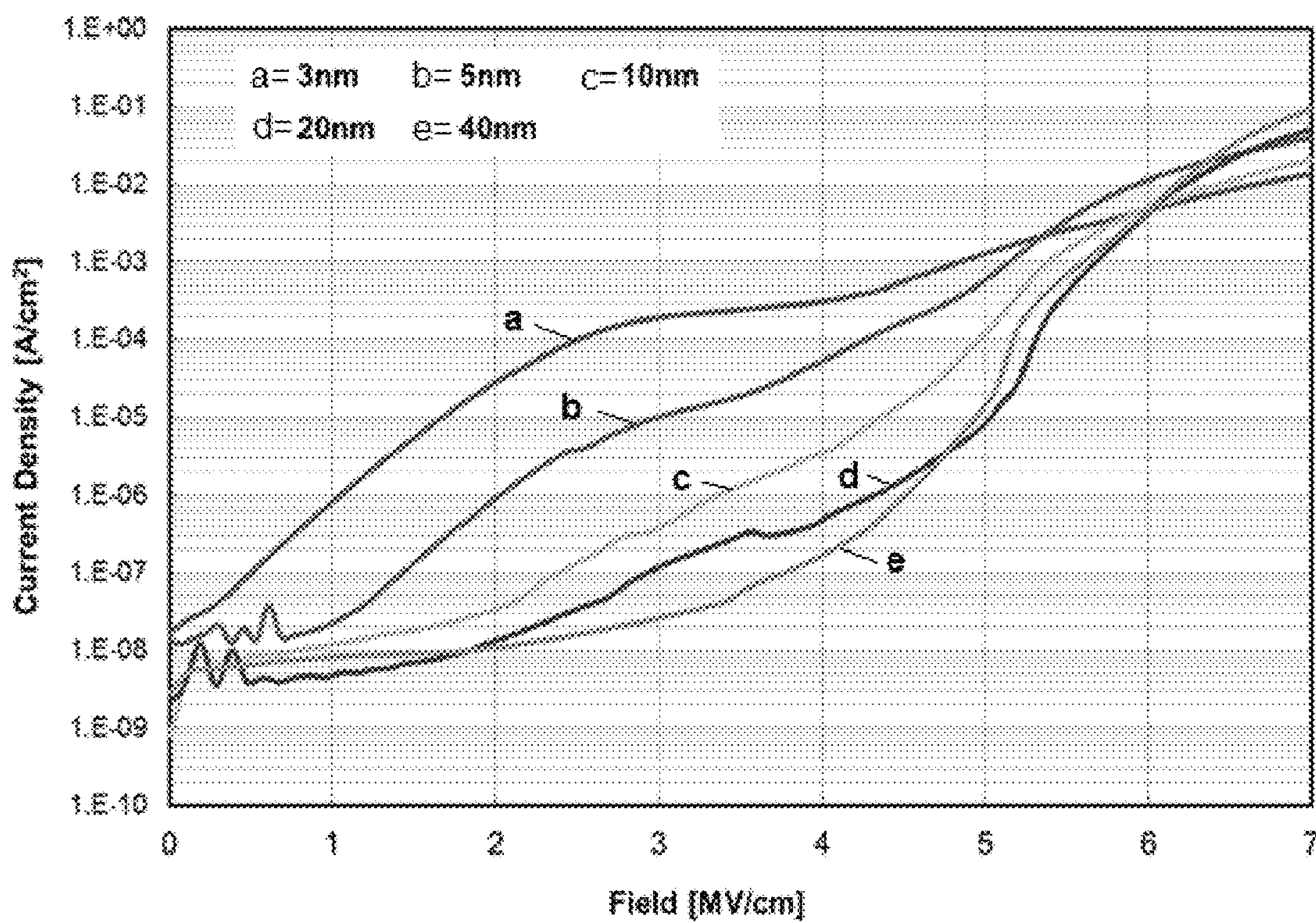
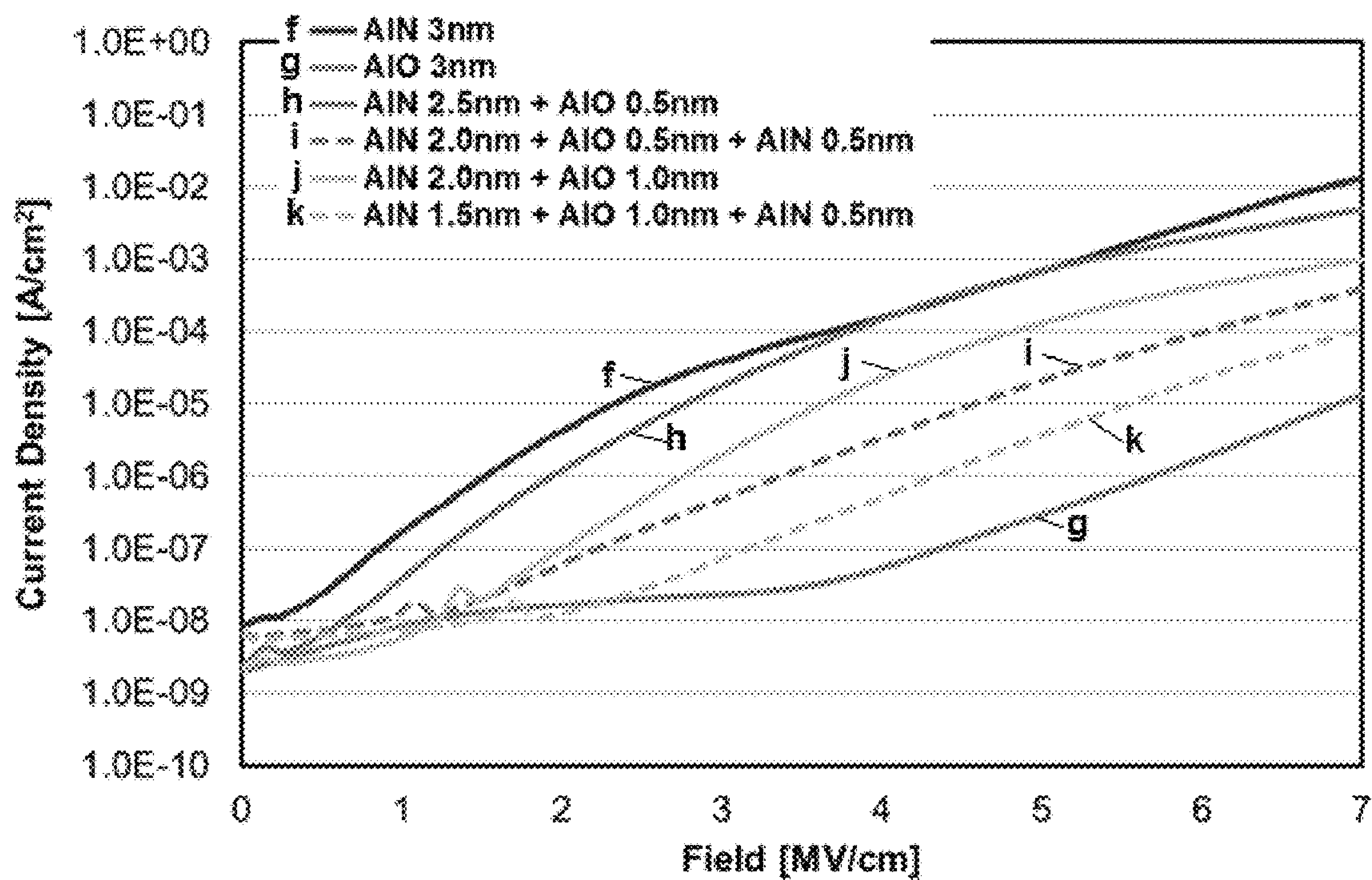


Fig. 4



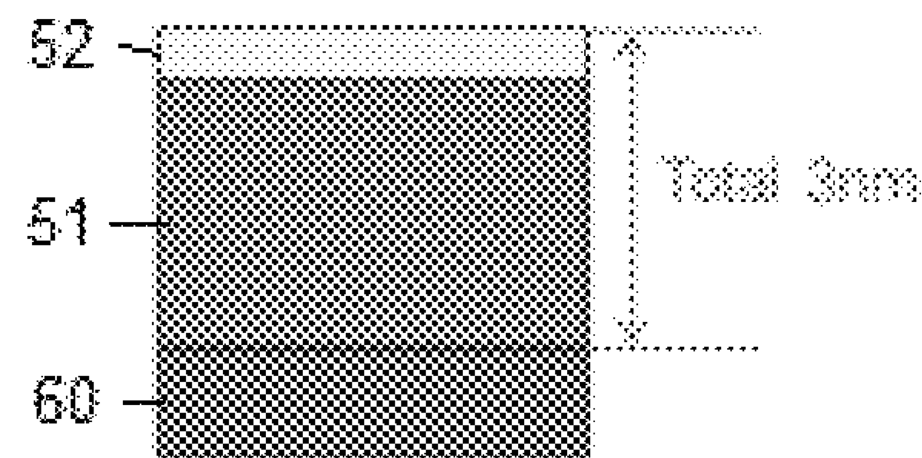


Fig. 5a

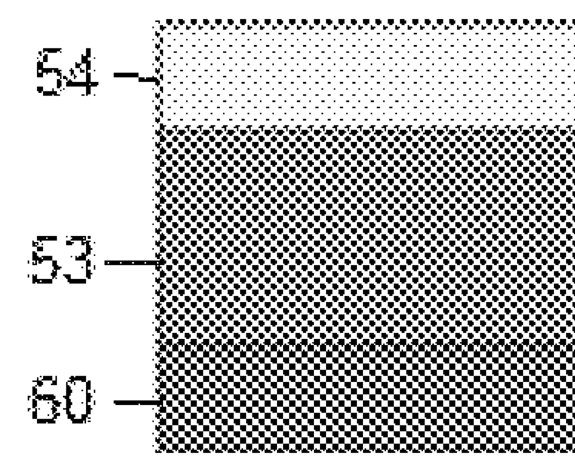


Fig. 5b

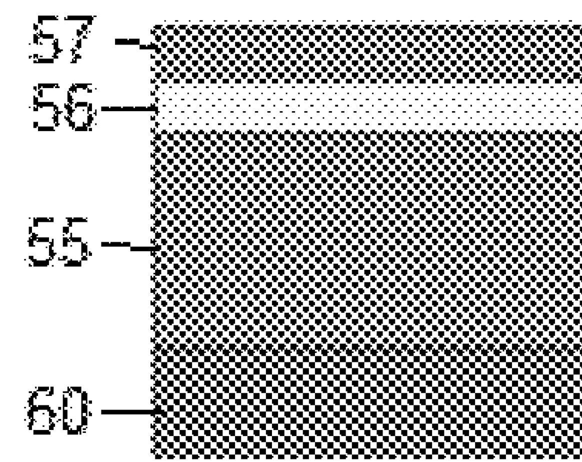


Fig. 5c

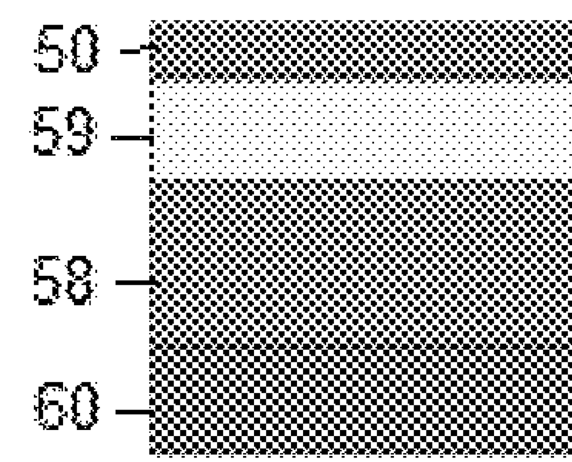


Fig. 5d

METHOD FOR FORMING ALUMINUM NITRIDE-BASED FILM BY PEALD

BACKGROUND

Field of the Invention

The present invention relates generally to a method for forming an aluminum nitride-based film on a substrate by plasma-enhanced atomic layer deposition (PEALD).

Related Art

Copper (Cu) has been used as a primary wiring material in multilevel interconnection of silicon devices. Since Cu easily diffuses to a transistor layer, an insulating layer, or the like, Ta/TaN or the like is used as a barrier metal layer facing the side and bottom of Cu wiring, and SiN, SiCN, or the like is typically used as a diffusion barrier layer and/or etch stop layer deposited on top of the Cu wiring.

In speed enhancement and energy savings of silicon devices, it is necessary to lower the effective dielectric constant of the devices themselves, and conventional diffusion barrier layers and etch step layers are not satisfactory. There are methods of lowering the effective dielectric constant. For example, by making the diffusion barrier layers and the etch stop layers thin, the effective dielectric constants of devices may be lowered. However, when the thickness of a film is in an extremely low range of 3 to 5 nm, the tunnel effect of electrons becomes more significant, and accordingly, the leakage current of the layers increases.

Aluminum nitride (AlN) is expected to replace SiCN used as a diffusion barrier layer and an etch stop layer. The present inventors have conducted research on leakage current improvement by an ultra thin film of AlN.

Any discussion of problems and solutions in relation to the related art has been included in this disclosure solely for the purposes of providing a context for the present invention, and should not be taken as an admission that any or all of the discussion was known at the time the invention was made.

SUMMARY

In some embodiments, a laminate constituted by an AlN layer and an AlO layer is formed by PEALD and replaces an AlN film. Surprisingly, as compared with the AlN film, the laminate can significantly improve leakage current even when the film is extremely thin such as that having a total thickness of about 3 nm. The laminate can be considered to be an aluminum oxynitride (AlON) film, but is an aluminum nitride-based film where AlN is a majority component of the laminate. Thus, the laminate can effectively be used as a diffusion barrier layer and/or etch stop layer, in place of an AlN film.

For purposes of summarizing aspects of the invention and the advantages achieved over the related art, certain objects and advantages of the invention are described in this disclosure. Of course, it is to be understood that not necessarily all such objects or advantages may be achieved in accordance with any particular embodiment of the invention. Thus, for example, those skilled in the art will recognize that the invention may be embodied or carried out in a manner that achieves or optimizes one advantage or group of advantages as taught herein without necessarily achieving other objects or advantages as may be taught or suggested herein.

Further aspects, features and advantages of this invention will become apparent from the detailed description which follows.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features of this invention will now be described with reference to the drawings of preferred embodiments which are intended to illustrate and not to limit the invention. The drawings are greatly simplified for illustrative purposes and are not necessarily to scale.

FIG. **1**A is a schematic representation of a PEALD (plasma-enhanced atomic layer deposition) apparatus for depositing a dielectric film usable in an embodiment of the present invention.

FIG. **1**B illustrates a schematic representation of a precursor supply system using a flow-pass system (FPS) usable in an embodiment of the present invention.

FIG. **2** illustrates a PEALD process sequence according to an embodiment of the present invention.

FIG. **3** is a graph showing leakage current properties of AlN films having various film thicknesses in Comparative Examples.

FIG. **4** is a graph showing leakage current properties of laminates of AlN/AlO layers having various film thicknesses in Examples according to embodiments of the present invention.

FIG. **5***a* to FIG. **5***d* illustrate layer structures used in the Examples.

DETAILED DESCRIPTION OF EMBODIMENTS

In this disclosure, "gas" may include vaporized solid and/or liquid and may be constituted by a single gas or a mixture of gases. In this disclosure, a process gas introduced to a reaction chamber through a showerhead may be comprised of, consist essentially of, or consist of a precursor gas and an additive gas. The precursor gas and the additive gas are typically introduced as a mixed gas or separately to a reaction space. The precursor gas can be introduced with a carrier gas such as a noble gas. The additive gas may be comprised of, consist essentially of, or consist of a reactant gas and a dilution gas such as a noble gas. The reactant gas and the dilution gas may be introduced as a mixed gas or separately to the reaction space. A precursor may be comprised of two or more precursors, and a reactant gas may be comprised of two or more reactant gases. The precursor is a gas chemisorbed on a substrate and typically containing aluminum element which constitutes a main structure of a matrix of a dielectric film, and the reactant gas for deposition is a gas reacting with the precursor chemisorbed on a substrate when the gas is excited to fix an atomic layer or monolayer on the substrate. "Chemisorption" refers to chemical saturation adsorption. A gas other than the process gas, i.e., a gas introduced without passing through the showerhead, may be used for, e.g., sealing the reaction space, which includes a seal gas such as a noble gas. In some embodiments, "film" refers to a layer continuously extending in a direction perpendicular to a thickness direction substantially without pinholes to cover an entire target or concerned surface, or simply a layer covering a target or concerned surface. In some embodiments, "layer" refers to a structure having a certain thickness formed on a surface or a synonym of film or a non-film structure. A film or layer may be constituted by a discrete single film or layer having certain characteristics or multiple films or layers, and a boundary between adjacent films or layers may or may not be clear and may be established based on physical, chemical, and/or any other characteristics, formation processes or sequence, and/or functions or purposes of the adjacent films or layers.

Further, in this disclosure, the article "a" or "an" refers to a species or a genus including multiple species unless specified otherwise. The terms "constituted by" and "having" refer independently to "typically or broadly compris-

ing", "comprising", "consisting essentially of", or "consisting of" in some embodiments. Also, in this disclosure, any defined meanings do not necessarily exclude ordinary and customary meanings in some embodiments.

Additionally, in this disclosure, any two numbers of a variable can constitute a workable range of the variable as the workable range can be determined based on routine work, and any ranges indicated may include or exclude the endpoints. Additionally, any values of variables indicated (regardless of whether they are indicated with "about" or not) may refer to precise values or approximate values and include equivalents, and may refer to average, median, representative, majority, etc. in some embodiments.

In the present disclosure where conditions and/or structures are not specified, the skilled artisan in the art can readily provide such conditions and/or structures, in view of the present disclosure, as a matter of routine experimentation. In all of the disclosed embodiments, any element used in an embodiment can be replaced with any elements equivalent thereto, including those explicitly, necessarily, or inherently disclosed herein, for the intended purposes. Further, the present invention can equally be applied to apparatuses and methods.

The embodiments will be explained with respect to preferred embodiments. However, the present invention is not limited to the preferred embodiments.

Some embodiments provide a method for forming an aluminum nitride-based film on a substrate by plasma-enhanced atomic layer deposition (PEALD) comprising: (a) forming at least one aluminum nitride (AlN) monolayer; and (b) forming at least one aluminum oxide (AlO) monolayer, said method comprising: (i) conducting step (a) at least once; (ii) conducting step (b) at least once, wherein steps (a) and (b) are alternately conducted continuously in this or reversed order without forming any other intervening layer therebetween so as to form as an AlN-based film a laminate constituted by an AlN layer and an AlO layer alternately deposited; and (iii) discontinuing steps (i) and (ii) before a total thickness of the laminate exceeds 10 nm, wherein a thickness of a portion constituted by the AlN layer is greater than a thickness of a portion constituted by the AlO layer. The word "discontinuing steps (i) and (ii)" refers to stopping or terminating steps (i) and (ii), immediately followed by a different step, without resuming step (i) or (ii), or without any further step concerning the laminate. The "aluminum nitride-based film" may also be referred to as a "laminated aluminum oxynitride (AlON) film," "oxygen-doped aluminum nitride film," "oxygen-diffused aluminum nitride film," "AlN/AlO stack multilayer film," "AlN/AlO bi-layer film," or the like, which can replace an aluminum nitride film in semiconductor devices. In some embodiments, the aluminum nitride-based film can suitably be used as a diffusion barrier film or etch stop film, without any post-deposition treatment such as heat treatment or annealing, or plasma-treatment, and without transferring a processed substrate to another reaction chamber. In some embodiments, step (i) and step (ii) are conducted in a same reaction chamber.

In some embodiments, leakage current can significantly be improved even when the total thickness of the laminate is 10 nm or less. Without being limited by a theory, this may be because electrons are trapped at an interface between an AlN layer and an AlO layer, thereby reducing leakage current, or because an AlN layer is oxidized and thus obtains leakage current properties of an AlO layer. When an AlO layer is deposited on an AlN layer, oxygen atoms penetrate the interface and diffuse or disperse into the AlN layer, and when an AlN layer is deposited on an AlO layer, nitrogen and/or hydrogen atoms penetrate the interface and diffuse or disperse into the AlO layer, thereby improving leakage current as a laminate. Considering the depth of penetration of oxygen atoms (or nitrogen and hydrogen atoms), in some embodiments, in step (iii), steps (i) and (ii) are discontinued before the total thickness of the laminate exceeds 5 nm (or 3 nm).

In some embodiments, the at least one AlN monolayer has a thickness of at least 0.2 nm (typically at least 0.5 nm), and the at least one AlO monolayer has a thickness of at least 0.2 nm (typically at least 0.5 nm), so that various layer structures of a laminate can be constructed, and film properties including leakage current properties can effectively be adjusted. In some embodiments, the at least one AlN monolayer has a thickness of 0.3 nm to 3 nm, and the at least one AlO monolayer has a thickness of 0.3 nm to 2 nm.

In some embodiments, an AlN monolayer is deposited first on and in contact with the substrate, so that oxidation of an underlying layer such as a copper wiring pattern of the substrate can be avoided. In other embodiments, an AlO monolayer is deposited first on and in contact with the substrate.

In embodiments where an AlN monolayer is deposited first, step (i) is conducted twice, and step (ii) is conducted once, so that the laminate is constituted by two AlN layers and one AlO layer interposed between the two AlN layers. In some embodiments, the substrate has a copper wiring pattern on its surface, and the AlN-based film is deposited on the copper wiring pattern as a diffusion blocking film or etch stop film. In some embodiments, the total number of layers of the laminate may be 2 to 20, 2 to 10, or 2 to 5.

In some embodiments, step (i) and step (ii) use a same aluminum-containing precursor for depositing the AlN layer and AlO layer. In other embodiments, step (i) and step (ii) use different aluminum-containing precursors for depositing the AlN layer and AlO layer.

In some embodiments, the precursor is one or more aluminum compounds containing one or two alkyl groups and one or two alkoxy or alkylamine groups in its molecule, wherein the total number of groups attached to aluminum is three, or three alkyl groups in its molecule. Alternatively, the aluminum compound may contain an alkynyl group and/or an alkyloxy or alkylamine group. In some embodiments, the aluminum compound is halogen-free. In some embodiments, the precursor is at least one aluminum compound selected from the group consisting of $Al(C_xH_y)_2(OC_zH_a)$, $Al(C_xH_y)(OC_zH_a)_2$, and $Al(C_xH_y)_2(NC_zH_a)$, wherein x, y, z, and a are integers. In some embodiments, x is two or more (e.g., 2 to 5), y is three or more (e.g., 3 to 11), z is three or more (e.g., 3 to 6), and a is five or more (e.g., 5 to 13). Typically, the aluminum compound includes $Al(CH_3)_3$ (TMA; trimethylaluminum), $Al(CH_3)_2(OC_3H_7)$, $Al(CH_3)(OC_3H_7)_2$, and $Al(CH_3)_2(NC_3H_7)$.

In some embodiments, the reactant gas for depositing an AlN monolayer is nitrogen gas and/or hydrogen gas, which is capable of generating a nitrogen and/or hydrogen plasma and is not thermally reactive to the precursor without a plasma. In some embodiments, the reactant gas for depositing an AlO monolayer is oxygen gas or an oxygen-containing gas such as ozone, nitrogen oxide (N_2O), etc., which is capable of generating an oxygen plasma and is not thermally reactive to the precursor without a plasma.

In some embodiments, the carrier gas and/or dilution gas is used which is at least one gas selected from rare gases such as Ar, He, Kr, and Xe.

In some embodiments, in step (a) and/or step (b), a monolayer is formed per process cycle comprising feeding

a precursor to a reaction space in a pulse and applying RF power to the reaction space, wherein the reactant gas flows continuously through the precursor feeding and the RF power application.

FIG. **2** illustrates a PEALD process sequence according to an embodiment of the present invention. In this disclosure, the width of each column does not necessarily represent the actual time length, and a raised level of the line in each row represents an ON-state whereas a bottom level of the line in each row represents an OFF-state. In this PEALD process, the process cycle includes the AlN deposition cycle and the AlO deposition cycle. The AlN deposition cycle includes steps of feeding precursor A to a reaction zone, purging the reaction zone, applying RF power to the reaction zone, and purging the reaction zone in this order, wherein a purge/carrier gas is supplied continuously to the reaction zone throughout the entire steps of deposition cycle, and reactant gas C for deposition is supplied continuously to the reaction zone throughout the entire steps of deposition cycle. In the AlN deposition cycle, steps of feeding precursor A, purging the reaction zone, applying RF power to the reaction zone, and purging the reaction zone can be repeated p times (p is an integer of 5 to 100, typically 8 to 50), depending on the target compositions and quality of the AlN film, although repeating is not required.

The AlO deposition cycle is conducted continuously after the AlN deposition cycle. The AlN deposition cycle includes steps of feeding precursor A to a reaction zone, purging the reaction zone, applying RF power to the reaction zone, and purging the reaction zone in this order, wherein a purge/carrier gas is supplied continuously to the reaction zone throughout the entire steps of deposition cycle, and reactant gas D for deposition is supplied continuously to the reaction zone throughout the entire steps of deposition cycle. In the AlO deposition cycle, steps of feeding precursor A, purging the reaction zone, applying RF power to the reaction zone, and purging the reaction zone can be repeated q times (q is an integer of 3 to 50, typically 5 to 20), depending on the target compositions and quality of the AlO film, although repeating is not required.

In this disclosure, the word "continuously" refers to at least one of the following: without breaking a vacuum, without being exposed to air, without opening a chamber, as an in-situ process, without interruption as a step in sequence, and without changing main process conditions, depending on the embodiment. In some embodiments, an auxiliary step such as a delay between steps or other step immaterial or insubstantial in the context does not count as a step, and thus, the word "continuously" does not exclude an intervening auxiliary step.

In the above, the AlN deposition cycle can be conducted after the AlO deposition cycle, and the deposition can be started with the AlO deposition cycle, in place of the AlN deposition cycle. In any event, the AlN deposition cycle and the AlO deposition cycle are conducted alternately, wherein "p" and "q" may be different at each corresponding cycle. In some embodiments, "p" and "q" and the number of each AlN film and AlO film are controlled so that the total thickness of the AlN film is greater than the total thickness of the AlO film. Further, precursor A need not be the same in the AlN deposition cycle and the AlO deposition cycle, and can be different, although the use of the same precursor is preferable. Also, two or more precursors can be used by supplying them as a single mixture gas or supplying them separately.

In the sequence illustrated in FIG. **2**, the precursor is supplied in a pulse using a carrier gas which is continuously supplied. This can be accomplished using a flow-pass system (FPS) wherein a carrier gas line is provided with a detour line having a precursor reservoir (bottle), and the main line and the detour line are switched, wherein when only a carrier gas is intended to be fed to a reaction chamber, the detour line is closed, whereas when both the carrier gas and a precursor gas are intended to be fed to the reaction chamber, the main line is closed and the carrier gas flows through the detour line and flows out from the bottle together with the precursor gas. In this way, the carrier gas can continuously flow into the reaction chamber, and can carry the precursor gas in pulses by switching the main line and the detour line. FIG. **1**B illustrates a precursor supply system using a flow-pass system (FPS) according to an embodiment of the present invention (black valves indicate that the valves are closed). As shown in (a) in FIG. **1**B, when feeding a precursor to a reaction chamber (not shown), first, a carrier gas such as Ar (or He) flows through a gas line with valves b and c, and then enters a bottle (reservoir) **20**. The carrier gas flows out from the bottle **20** while carrying a precursor gas in an amount corresponding to a vapor pressure inside the bottle **20**, and flows through a gas line with valves f and e, and is then fed to the reaction chamber together with the precursor. In the above, valves a and d are closed. When feeding only the carrier gas (noble gas) to the reaction chamber, as shown in (b) in FIG. **1**B, the carrier gas flows through the gas line with the valve a while bypassing the bottle **20**. In the above, valves b, c, e, and fare closed.

In some embodiments, the AlN and AlO deposition cycles may be performed by PEALD, one cycle of which is conducted under conditions shown in Table 1 below.

TABLE 1

(the numbers are approximate)
Conditions for Deposition Cycle

Substrate temperature	100 to 400° C. (preferably 300 to 400° C.)
Pressure	300 to 500 Pa (preferably 400 to 450 Pa)
Precursor pulse	0.1 to 0.5 sec (preferably 0.1 to 0.3 sec)
Precursor purge	0.3 to 1.0 sec (preferably 0.3 to 0.5 sec)
Flow rate of reactant	500 to 3000 sccm (preferably 2000 to 3000 sccm) for N_2 and O_2; 50 to 300 sccm (preferably 50 to 200 sccm) for H_2
Carrier gas	500 to 3000 sccm (typically 2000 sccm)
Dilution gas	500 to 2000 sccm (typically 1000 sccm) when O_2 or other oxidizing gas is used
RF power (13.56 MHz) for a 300-mm wafer	100 to 400 W (preferably 200 to 300 W)
RF power pulse	0.5 to 2.0 sec (preferably 1.0 to 1.5 sec)
Purge	0.1 to 0.3 sec (preferably 0.1 to 0.2 sec)
Growth rate per cycle	0.06 to 0.07 nm/cycle

The precursor may be provided with the aid of a carrier gas. Since ALD is a self-limiting adsorption reaction process, the number of deposited precursor molecules is determined by the number of reactive surface sites and is independent of the precursor exposure after saturation, and a supply of the precursor is such that the reactive surface sites are saturated thereby per cycle. A plasma for deposition may be generated in situ, for example, in an ammonia gas that flows continuously throughout the deposition cycle. In other embodiments the plasma may be generated remotely and provided to the reaction chamber.

As mentioned above, each pulse or phase of each deposition cycle is preferably self-limiting. An excess of reactants is supplied in each phase to saturate the susceptible structure surfaces. Surface saturation ensures reactant occupation of all available reactive sites (subject, for example, to physical size or "steric hindrance" restraints) and thus

ensures excellent step coverage. In some embodiments the pulse time of one or more of the reactants can be reduced such that complete saturation is not achieved and less than a monolayer is adsorbed on the substrate surface.

The process cycle can be performed using any suitable apparatus including an apparatus illustrated in FIG. **1**A, for example. FIG. **1**A is a schematic view of a PEALD apparatus, desirably in conjunction with controls programmed to conduct the sequences described below, usable in some embodiments of the present invention. In this figure, by providing a pair of electrically conductive flat-plate electrodes **4**, **2** in parallel and facing each other in the interior **11** (reaction zone) of a reaction chamber **3**, applying HRF power (13.56 MHz or 27 MHz) **20** to one side, and electrically grounding the other side **12**, a plasma is excited between the electrodes. A temperature regulator is provided in a lower stage **2** (the lower electrode), and a temperature of a substrate **1** placed thereon is kept constant at a given temperature. The upper electrode **4** serves as a shower plate as well, and reactant gas (and noble gas) and precursor gas are introduced into the reaction chamber **3** through a gas line **21** and a gas line **22**, respectively, and through the shower plate **4**. Additionally, in the reaction chamber **3**, a circular duct **13** with an exhaust line **7** is provided, through which gas in the interior **11** of the reaction chamber **3** is exhausted. Additionally, a transfer chamber **5** disposed below the reaction chamber **3** is provided with a seal gas line **24** to introduce seal gas into the interior **11** of the reaction chamber **3** via the interior **16** (transfer zone) of the transfer chamber **5** wherein a separation plate **14** for separating the reaction zone and the transfer zone is provided (a gate valve through which a wafer is transferred into or from the transfer chamber **5** is omitted from this figure). The transfer chamber is also provided with an exhaust line **6**. In some embodiments, the deposition of multi-element film and surface treatment are performed in the same, so that all the steps can continuously be conducted without exposing the substrate to air or other oxygen-containing atmosphere. In some embodiments, a remote plasma unit can be used for exciting a gas.

In some embodiments, in the apparatus depicted in FIG. **1**A, the system of switching flow of an inactive gas and flow of a precursor gas illustrated in FIG. **1**B (described earlier) can be used to introduce the precursor gas in pulses without substantially fluctuating pressure of the reaction chamber.

In some embodiments, a dual chamber reactor (two sections or compartments for processing wafers disposed closely to each other) can be used, wherein a reactant gas and a noble gas can be supplied through a shared line whereas a precursor gas is supplied through unshared lines.

A skilled artisan will appreciate that the apparatus includes one or more controller(s) (not shown) programmed or otherwise configured to cause the deposition and reactor cleaning processes described elsewhere herein to be conducted. The controller(s) are communicated with the various power sources, heating systems, pumps, robotics and gas flow controllers or valves of the reactor, as will be appreciated by the skilled artisan.

The present invention is further explained with reference to working examples below. However, the examples are not intended to limit the present invention. In the examples where conditions and/or structures are not specified, the skilled artisan in the art can readily provide such conditions and/or structures, in view of the present disclosure, as a matter of routine experimentation. Also, the numbers applied in the specific examples can be modified by a range of at least ±50% in some embodiments, and the numbers are approximate.

EXAMPLES

An aluminum nitride/oxide film was formed on a Si substrate (Φ300 mm) by PEALD, one cycle of which was conducted as illustrated in FIG. **2** under the conditions shown in Table 2 below using the PEALD apparatus illustrated in FIG. **1**A (including a modification illustrated in FIG. **1**B). The number of each AlN and AlO cycles in each example (Examples a to k) is shown in Table 3. In the examples, TMA (trimethylaluminum) was used as a precursor.

TABLE 2

(the numbers are approximate)

Process		AlN	AlO
Precurser		TMA	TMA
Precurser Bottle Temp.	[deg. C.]	35	35
Carrier Ar (/RC)	[sccm]	2000	2000
Dilution Ar (/RC)	[sccm]	—	1000
N2 (/RC)	[sccm]	3000	—
H2 (/RC)	[sccm]	100	—
O2 (/RC)	[sccm]	—	2000
Seal Ar (/RC)	[sccm]	200	200
RC Press	[Pa]	400	300
RF (13.56 MHz)	[W]	300	—
process temp.	[deg. C.]	300	300
	TMA feed	0.1	0.1
	TMA purge	0.3	0.5
Deposition Cycle Time [sec]	RF On	1.0	1.0
	Post purge	0.1	0.1
	Total	1.5	1.7

TABLE 3

Example	Layer structure	1st AlN cycle	AlO cycle	2nd AlN cycle
a	AlN 3 nm	50	0	0
b	AlN 5 nm	70	0	0
c	AlN 10 nm	140	0	0
d	AlN 20 nm	280	0	0
e	AlN 40 nm	560	0	0
f	AlN 3 nm	50	0	0
g	AlO 3 nm	0	27	0
h	AlN 2.5 nm + AlO 0.5 nm	35	5	0
i	AlN 2.0 nm + AlO 0.5 nm + AlN 0.5 nm	30	5	10
j	AlN 2.0 nm + AlO 1.0 nm	30	9	0
k	AlN 1.5 nm + AlO 1.0 nm + AlN 0.5 nm	25	9	10

FIG. **5***a* to FIG. **5***d* illustrate layer structures used in Examples h (FIG. **5***a*), i (FIG. **5***b*), j (FIG. **5***c*), and k (FIG. **5***d*). In Example h (FIG. **5***a*), on a substrate **60**, an AlN film **51** with a thickness of 2.5 nm, and an AlO film **52** with a thickness of 0.5 nm were deposited in this order. In Example i (FIG. **5***b*), on a substrate **60**, an AlN film **53** with a thickness of 2.5 nm, and an AlO film **54** with a thickness of 1.0 nm were deposited in this order. In Example j (FIG. **5***c*), on a substrate **60**, a 1st AlN film **55** with a thickness of 2.5 nm, an AlO film **56** with a thickness of 0.5 nm, and a 2nd AlN film **57** with a thickness of 0.5 nm were deposited in this order. In Example k (FIG. **5***d*), on a substrate **60**, a 1st AlN film **58** with a thickness of 1.5 nm, an AlO film **59** with a thickness of 1.0 nm, and a 2nd AlN film **50** with a thickness of 0.5 nm were deposited in this order.

The obtained film was analyzed in terms of leakage current. The results are shown in FIGS. **3** and **4**. As shown in FIG. **3**, when the thickness of the AlN film was as thick

as 20 to 40 nm (Examples d, e), leakage current did not increase significantly. However, when the thickness of the AlN film was as thin as 10 nm (Example c), leakage current started to increase. When the thickness of the AlN film was as thin as 3 to 5 nm (Examples a, b), leakage current significantly increased particularly when the thickness was 3 nm (Example a).

FIG. **4** shows Examples f to k wherein the total thickness of each film was controlled at 3 nm. As shown in FIG. **4**, when the AlN film was used alone (Example f), leakage current significantly increased, as compared with when the AlO film was used alone (Example g). Although AlO films exhibit good leakage current, AlO films are not usable in applications where an underlying layer such as copper wiring pattern is easily oxidized. When a laminate was constituted by an AlN film of 2.5 nm and an AlO film of 0.5 nm (Example h), leakage current was slightly improved; however, when a laminate was constituted by a 1st AlN film of 2.0 nm, an AlO film of 0.5 nm, and 2nd AlN film (Example i), leakage current was significantly improved, although the thickness of the AlO film was the same (0.5 nm), and the total thickness of the laminate was the same (3 nm). This indicates that by inserting the AlO film between the AlN films, i.e., by providing two interfaces (Example i), oxygen diffusion and nitrogen/hydrogen diffusion progressed more through the interfaces than when only one interface was provided (Example h), thereby improving leakage current. When the thickness of an AlO film was increased to 1.0 nm without a 2nd AlN film (Example j), leakage current was improved as compared with leakage current of the laminate wherein the thickness of an AlO film was 0.5 nm without a 2nd AlN film (Example h). However, because the laminate of Example j did not have a 2nd AlN film, despite the fact that the thickness of the AlO film was twice the thickness of the AlO film of Example i, leakage current was not as good as that in Example i with a 2nd AlN film. When the thickness of an AlO film was 1.0 nm with a 2nd AlN film (Example k), leakage current was most significantly improved.

It will be understood by those of skill in the art that numerous and various modifications can be made without departing from the spirit of the present invention. Therefore, it should be clearly understood that the forms of the present invention are illustrative only and are not intended to limit the scope of the present invention.

I claim:

1. A method for forming an aluminum nitride-based film on a substrate by plasma-enhanced atomic layer deposition (PEALD) comprising: (a) forming at least one aluminum nitride (AlN) monolayer; and (b) forming at least one aluminum oxide (AlO) monolayer, said method comprising:

(i) conducting step (a) at least once;

(ii) conducting step (b) at least once, wherein steps (a) and (b) are alternately conducted continuously in this or reversed order without forming any other intervening layer therebetween so as to form as an AlN-based film a laminate constituted by an AlN layer and an AlO layer alternately deposited; and

(iii) terminating steps (i) and (ii) before a total thickness of the laminate exceeds 10 nm, wherein a thickness of a portion constituted by the AlN layer is greater than a thickness of a portion constituted by the AlO layer,

wherein the substrate has a copper wiring pattern on its surface, and the AlN-based film is deposited on the copper wiring pattern as a diffusion-blocking film or etch stop film.

2. The method according to claim **1**, wherein in step (iii), steps (i) and (ii) are discontinued before the total thickness of the laminate exceeds 5 nm.

3. The method according to claim **1**, wherein the at least one AlN monolayer has a thickness of 0.3 nm to 3 nm, and the at least one AlO monolayer has a thickness of 0.3 nm to 2 nm.

4. The method according to claim **1**, wherein the at least one AlN monolayer is deposited on and in contact with the substrate.

5. The method according to claim **4**, wherein step (i) is conducted twice, and step (ii) is conducted once, so that the laminate is constituted by two AlN layers and one AlO layer interposed between the two AlN layers.

6. The method according to claim **1**, wherein step (i) and step (ii) are conducted in a same reaction chamber.

7. The method according to claim **1**, wherein step (i) and step (ii) use a same aluminum-containing precursor for depositing the AlN layer and AlO layer.

8. The method according to claim **1**, wherein step (i) uses N_2 and H_2 as a reactant gas.

9. The method according to claim **8**, wherein in step (a), a monolayer is formed per process cycle comprising feeding a precursor to a reaction space in a pulse and applying RF power to the reaction space, wherein the reactant gas flows continuously through the precursor feeding and the RF power application.

10. The method according to claim **1**, wherein step (ii) uses O_2 or N_2O as a reactant gas.

11. The method according to claim **10**, wherein in step (b), a monolayer is formed per process cycle comprising feeding a precursor to a reaction space in a pulse and applying RF power to the reaction space, wherein the reactant gas flows continuously through the precursor feeding and the RF power application.

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